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H4H HAX2 H100 H104 H105 H121 H140 H152 H157 H170 H175 H212 H221 H222 H231 H232 H233 H234 H251

(56) Documents cited
WO 89/04108 A FR 002559886 A US 5065131 A
US 4347431 A US 4135053 A

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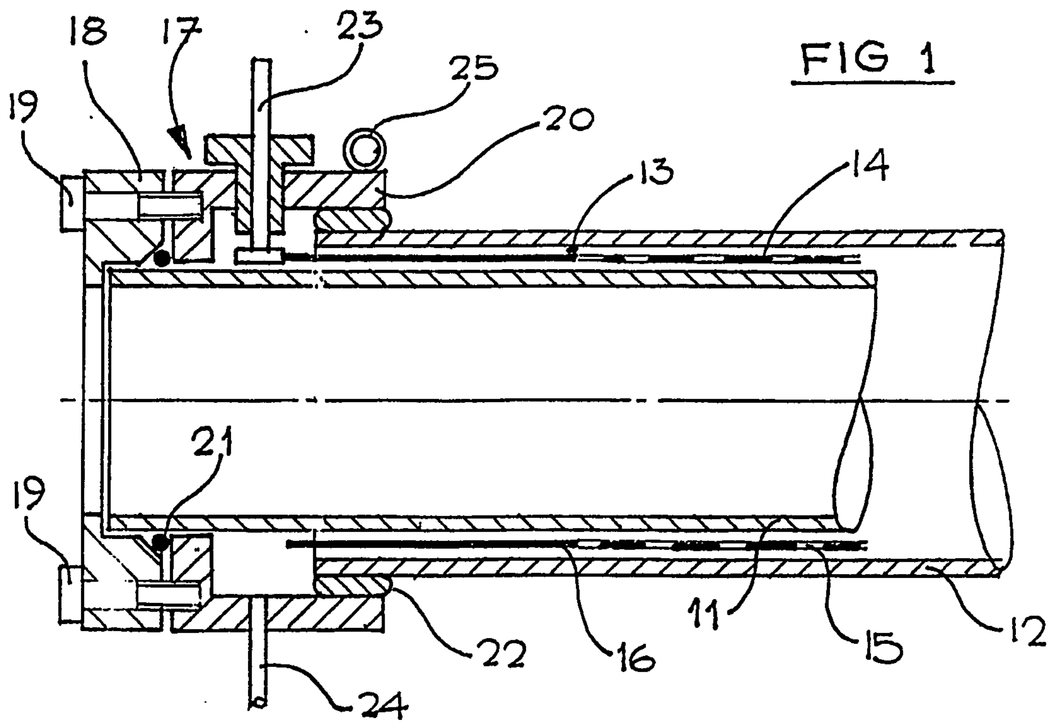
UK CL (Edition K) H5H HAA3 HAX2
INT CL[5] H05B 3/44 3/62 3/64
Online databases: WPI, CLAIMS

(54) Inert gas protects carbon heating element

(57) Electrically-powered heater units for use in furnaces requiring an internal temperature of 1500°C and above employ graphite heater elements 14, 15 enclosed within a sealed double-walled support 11, 12 of heat-conductive ceramic material. The element-containing enclosed volume of the double-walled heater unit is continuously purged with an inert gas such as nitrogen e.g. through inlet pipe 24 and an outlet pipe not shown. The ceramic is preferably alumina. The graphite may be coated with a nitride. The inert gas and the coating prevent the ceramic oxidising the carbon.

FIG 1



At least one drawing originally filed was informal and the print reproduced here is taken from a later filed formal copy.

The claims were filed later than the filing date within the period prescribed by Rule 25(1) of the Patents Rules 1990.

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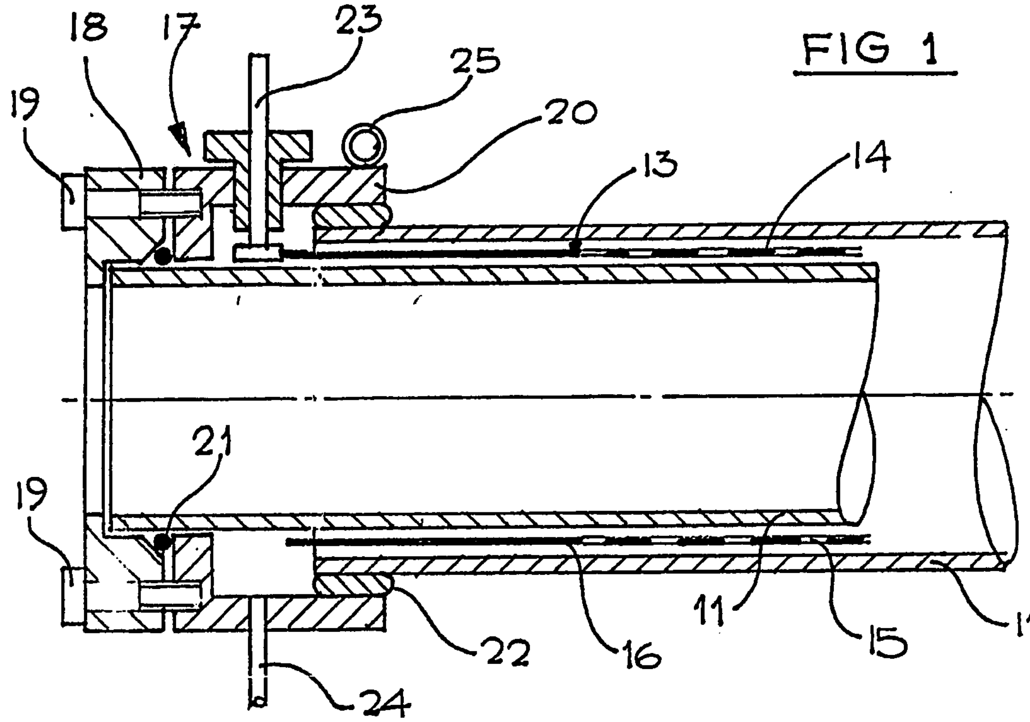
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FIG 1

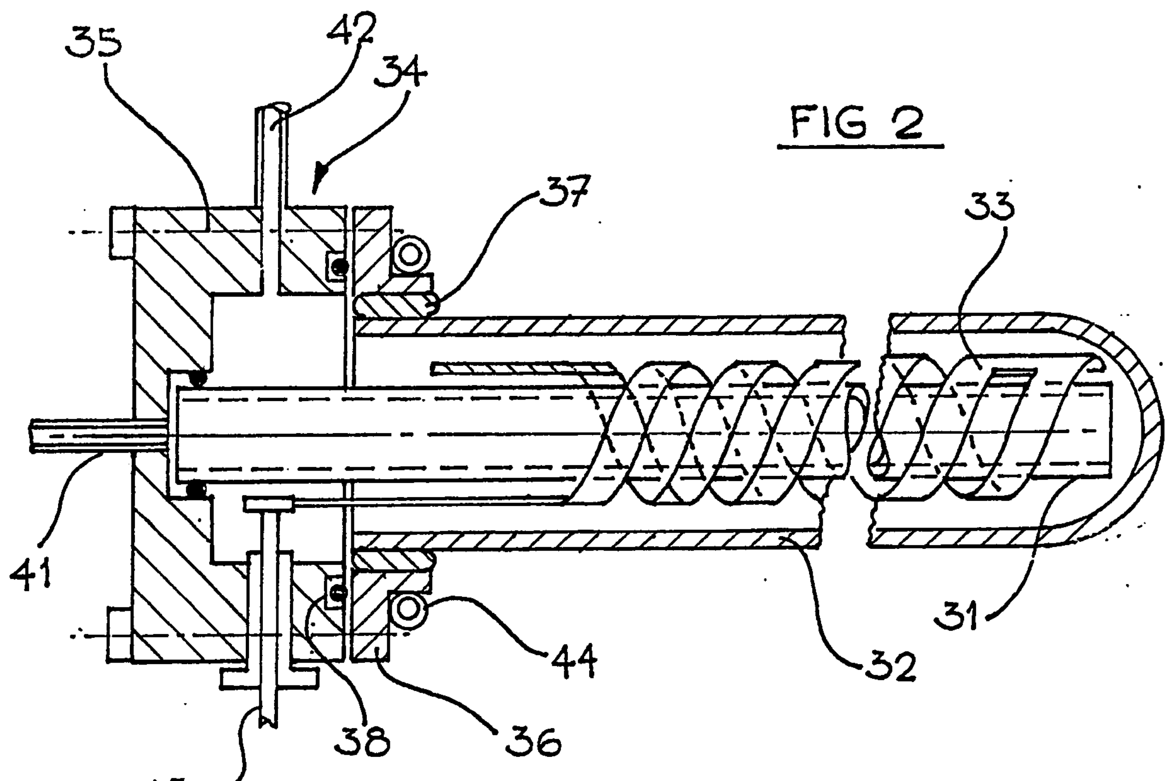
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FIG 2

Heaters

This invention relates to heaters, and concerns in particular electrically-powered heater units suitable for use in furnaces of the sort requiring an internal temperature of 1500°C and above.

In a number of technical processes, such as the preparation of crystals of high purity, or the deposition of layers of material (such as a semi-conductor device component) upon some substrate, there is a need for the heating of an object, or of a source of metal to provide a vapour, to a relatively high temperature, typically 1500°C, and even 1700°C, and above. There are various problems in designing and building an electric furnace suited for this purpose, one of which is the choice of an appropriate heating element and the substrate/former upon which it is mounted. The parameters which determine the choice are many and varied, but two important ones are the atmosphere to which the element is to be exposed when hot, and the chemical nature of the substrate/former carrying it; clearly, the heater element must be impervious to the former and unaffected by the latter.

Much of the time the furnace will be operating while containing either a vacuum or a non-reactive, and especially a non-oxidising, atmosphere, and it is acceptable to employ as the heater element a material, such as graphite or graphitic carbon, that is oxidisable, the element simply lining the inside walls of the furnace enclosure. At other times, however, the in-use atmosphere of the furnace is such that there must

be employed an oxygen-resistant element material. Typical such materials are molybdenum disilicide, silicon carbide, and iron-chrome-aluminium (all of which can be employed in a number of different physical/structural forms); these work satisfactorily in air or other oxidising atmospheres at temperatures up to 1500°, 1650° and 1325° respectively, but are not entirely satisfactory when operated in non-oxidising atmospheres such as carborising or reducing atmospheres, or in vacuum.

It might be thought that one solution to this problem was to separate the heater element from the furnace atmosphere (or absence thereof) by building the element into a sealed heater unit where the element is held enclosed within a sealed double-walled support of some suitable heat-conductive material - typically an impervious ceramic such as alumina (aluminium oxide), zirconia (zirconium oxide), beryllia (beryllium oxide) or thoria (thorium oxide) - but even then there is a problem, for at the temperatures attained, in the region of 1700°C, the ceramic itself is an oxidiser, and a heating element made from an oxidisable material, such as graphite, has a short but spectacular life.

It has now been found, however, that for certain electrical heating element materials, and particularly for those constructed from graphite, this difficulty can be overcome, in a simple but nevertheless surprisingly effective manner, by continuously purging the element-containing enclosed volume of the double-walled heater unit with an inert gas. In particular, it has been observed that if a graphite element be disposed enclosed within a double-walled ceramic support, and in use the enclosure is constantly purged with an inert gas such as nitrogen, the unit may be used at temperatures up to

1700°C and slightly above without any significant deterioration in the element *per se*.

In one aspect, therefore, the invention provides a heater unit comprising:-

two spaced walls each of an impervious ceramic material joined one to the other around the periphery thereof to make a sealed enclosure;

sealed within the enclosure, a graphite-based heating element;

lead means to supply electrical power to the heating element; and

port means by which purging inert gas can be supplied to, and withdrawn from, the sealed enclosure.

The invention provides a heater unit - that is to say, a unit that when appropriately provided with electrical power gets hot, rather like the bar of an electric fire. As will become apparent, the unit may take any suitable physical shape - a flat plate-like device, for example - but is preferably a tubular device, especially a hollow one in which material to be heated thereby can be passed along the middle of the tube.

The heater unit comprises two spaced walls joined one to the other around the periphery thereof to make a sealed enclosure. The walls may have any physical shape and form appropriate to the desired shape and form of the heater unit, save that they must be sealably joinable (around the "common" periphery) so that there can be formed a sealed enclosure (this sealing may be

accomplished in any suitable manner). Although each wall might have a plate-like form, the two joined together forming a thicker plate-like heater unit, it is in fact preferred if each be an elongate tube (and conveniently a circular cross-section tube), one smaller in section than the other, the two being disposed one co-axially within the other to form an elongate annular enclosure within which the element is disposed. In such a co-axial structure the tube ends are sealed, inner to outer, conveniently with end caps (typically of brass, aluminium or stainless steel), to provide the desired sealed enclosure.

The walls are sealed one to another, and this may be effected in any appropriate manner. In the case of a tubular unit with end caps it is convenient to seal the cap to the outer tubular wall with a silicone rubber seal, but since the usable silicone materials are not especially heat stable it may be desirable to provide cooling of the end cap adjacent the seal to prevent the seal overheating and being destroyed. The embodiments described hereinafter with reference to the accompanying Drawings employ end cap cooling for this purpose.

The enclosure walls are made of an impervious ceramic material, such as one of those mentioned above, and especially re-crystallised alumina (because it is relatively cheap, non-toxic, and a good electrical insulator while also a good thermal conductor). A typical such material is that available under the name PUROX from Morgan Ceramics or ZYALOX 998 from Vesuvius.

The size of the enclosure walls (and thus of the heater unit), and the thickness thereof, can be any thought suitable for the application at hand. For example, a common tubular unit might be 90 cm long and 7.5 cm external diameter, the walls themselves being

0.6 cm thick, and the between-wall space being 0.5 cm across.

Sealed within the enclosure is a graphite-based heating element. Graphite is an excellent material for use in the construction of heating elements, being both relatively cheap and easy to shape, and graphite or graphitic carbon formed in a sheet, physically rather like thin paper card, is a particularly useful type of graphite to employ, for it is extremely easy to shape and deploy, as well as having electrical and thermal characteristics which make it ideal for use as an electrical resistance heating element. An especially suitable variety of "paper-like" graphite is that obtainable under the name FLEXICARB from Flexitalic. It is available in sheets from 0.2 to 1.5 mm thick having electrical resistivity of 10 $\Omega.mm^2/m$ in the plane of the sheet and 600 $\Omega.mm^2/m$ perpendicular thereto (the thermal conductivity is 200 W/m.°C in the plane, and 7 W/m.°C perpendicularly), and can easily and simply be cut into any shape and size required.

The paper-like graphite preferred for use as the heating element material is not particularly chemically inert, although by surrounding it with an atmosphere of inert gas its life can be significantly prolonged. However, that life can be improved still further if prior to use there is deposited upon the element a thin coating or layer of a nitride (or other ceramic) such as boron nitride. A useful such coating is about 0.1 micrometre thick, and may be formed by standard chemical vapour deposition techniques.

The heating element may take any suitable physical form, the form naturally being appropriate to both the structure of the unit (whether flat of tubular, for instance) and the purpose of the unit (thus, whether it is to be a rod unit heating what is outside it, or a tube unit heating what is passed through it, and what temperature distribution is required). For a tubular unit one typical form of heater element is that of a helix, wound around the inner tube as a former, while another is of a plurality of linear bars, either running axially parallel with or transversely of the tube. Which form is chosen may depend upon the temperature distribution required. An element with axially parallel bars can assist in levelling out, and making uniform, the temperature along the unit, while one with transverse bars - and particularly bars which are not all of the same width or spacing - can be employed to give a distinct temperature profile along the unit.

Lead means are provided to supply electrical power to the heating element. The lead means can be, quite simply, suitable electrical connections from the world outside the enclosure to the element within, through the sealing between the walls (in a tubular unit, through the sealing end caps). However, because of the potentially very large temperature gradient between the connection means and the element itself, it is desirable to form the connections of some suitable material - copper, for example, or molybdenum - that has a similar thermal coefficient of expansion.

Inert gas is used to purge the enclosure of any oxidising environment, and naturally there are provided port means by which the purging inert gas can be

supplied to, and withdrawn from, the sealed enclosure. These port means may take any suitable form - with the tubular unit preferred they are conveniently pipes sealed into and through the sealing end caps, and connectable to a source and a drain of the gas.

The inert gas employed to do the purging may in effect be any gas not in itself reactive with the materials of the unit. It may be a true inert gas - that is, helium, argon, and so on - but most conveniently (mainly for cost reasons) it is nitrogen (which, though not strictly inert, does not react with any of the components under the conditions of use, and serves satisfactorily to purge - ie, sweep out - any oxidising vapour material within the enclosure, whether there from the start or formed therein during operation of the unit. In operation the inert gas may be employed at ambient (that is, atmospheric) pressure, and at a through rate of from 0.1 to 1 volumes (of the enclosure) per minute.

The heater unit of the invention, especially in its tubular forms, finds many uses in both small and large furnaces. Muffle-type (through-flow) units are particularly suited for use in small furnaces, of around 10 litre in volume, while the rod-type units are for the larger furnaces, such as 1000 litre and beyond, where a multiplicity of units is required uniformly to heat the enclosed volume. Even at temperatures in excess of 1700°C the heater unit of the invention has a relatively long life despite employing an inexpensive and rather reactive heating element made from graphite.

Two embodiments of the invention are now described, though by way of illustration only, with reference to the accompanying diagrammatic Drawings in which:

<u>Figure 1</u> shows an axial cross section through one end of a heater unit of the invention (the other end corresponds); and

<u>Figure 2</u> shows an axial cross section through another heater unit of the invention.

The heater unit of Figure 1 - only one end is shown; the other end corresponds - is a "muffle" tube - that is, it is of the large tubular type where the material to be heated is passed down the inside of the tube. It comprises an alumina inner tube (11) and co-axial therewith and spaced therefrom a slightly shorter alumina outer tube (12), the two defining an enclosure (13) within which is disposed a paper-like graphite heater element (generally 14) composed of a number of parallel bars (as 15) extending between two rings (as 16; only one is shown). An end cap (generally 17) has a stainless steel inner tube sealing portion (18; it is sealed to the inner tube 11 by a VITON O-ring 21) secured by bolts (as 19) to a brass outer tube sealing portion (20). It seals the two ends of the tubes 11, 12 one to the other (to close off the enclosure 13); the two end cap portions are also sealed one to the other by the sealing O-ring 21, while the second cap portion 20 is secured to the outside of the end of the outer tube 12 by a larger silicone rubber seal (22).

The outer tube end cap portion 20 extends some way beyond the actual tube end, and through the "overhanging" part pass both the (insulated) electrical

leads (23; the other lead is at the other end, not shown) to the element 14 and also the purging gas supply pipe (24; the exit is at the other end, not shown).

Finally, mounted (by soldering or brazing) onto the outer surface of the end cap portion 20, adjacent the silicone seal 22, is a water cooling pipe (25; not shown in detail). Another like cooling pipe is adjacent the seal at the other end (not shown).

The device of Figure 2 is more rod-like than that of Figure 1, and is intended to be inserted into a volume the temperature of which is to be raised.

The unit comprises a rod-like but nevertheless tubular inner tube (31) co-axially within a larger diameter, but shorter, outer tube or sheath (32), each of re-crystallised alumina. Wound helically around the outside of the inner tube 31, in the space enclosed between the inner and outer tubes 31/32, is a paper-like graphite heater element (33), and the enclosure is sealed off by a two-part end cap (generally 34) having an inner-tube sealing portion (35) and an outer-tube sealing portion (36) secured together (the outer-tube portion 36 is a short flanged tube; it is sealed to the outer-tube end by a large silicone seal 37 and to the inner-tube portion 35 by an O-ring 38).

In this embodiment the purging gas is fed to an axial inlet (41), whence it travels the length of the inner tube 31, returning along the enclosure between the two tubes and out *via* an exit (42) projecting sideways of the inner tube end cap sealing portion 35. Also disposed transaxially of this portion are the leads (43) to and from the element 33, while adjacent the silicone rubber seal 37 is mounted water cooling means (44).

CLAIMS

1. A heater unit comprising:-

two spaced walls each of an impervious ceramic material joined one to the other around the periphery thereof to make a sealed enclosure;

sealed within the enclosure, a graphite-based heating element;

lead means to supply electrical power to the heating element; and

port means by which purging inert gas can be supplied to, and withdrawn from, the sealed enclosure.

2. A heater unit as claimed in Claim 1 which is a tubular device.

3. A heater unit as claimed in Claim 2 which is a hollow tubular device in which material to be heated thereby can be passed along the middle of the tube.

4. A heater unit as claimed in any of the preceding Claims, wherein each wall is an elongate tube, one smaller in section than the other, the two being disposed one co-axially within the other to form an elongate annular enclosure within which the element is disposed.

5. A heater unit as claimed in Claim 4, wherein the tube ends are sealed, inner to outer, with end caps to provide the desired sealed enclosure.

6. A heater unit as claimed in Claim 5, wherein each end cap is sealed to the outer tubular wall, and there is provided cooling of the end cap adjacent the seal to prevent the seal overheating and being destroyed.

7. A heater unit as claimed in any of the preceding Claims, wherein the enclosure walls are made of re-crystallised alumina.

8. A heater unit as claimed in any of the preceding Claims, wherein the graphite-based heating element is in the form of a sheet, sized and shaped as required.

9. A heater unit as claimed in any of the preceding Claims, wherein the graphite-based heating element has thereover a thin coating or layer of a nitride (or other ceramic).

10. A heater unit as claimed in any of the preceding Claims, wherein the heating element for a tubular unit takes the form a helix, wound around the inner tube as a former, or of a plurality of linear bars, either running axially parallel with or circumferentially of the tube.

11. A heater unit as claimed in any of the preceding Claims, wherein the lead means provided to supply electrical power to the heating element are suitable electrical connections from the outside to the element within, the connections being of some suitable material that has a similar thermal coefficient of expansion.

12. A heater unit as claimed in any of the preceding Claims, wherein the port means by which the purging inert gas can be supplied to, and withdrawn from, the sealed enclosure, takes (for a tubular unit with end caps) the form of pipes sealed into and through the sealing end caps, and connectable to a source and a drain of the gas.

13. A heater unit as claimed in any of the preceding Claims and substantially as described hereinbefore.

14. A furnace whenever fitted with a heater unit as claimed in any of the preceding Claims.

Amendments to the claims have been filed as follows

1. A heater unit comprising:-

two spaced walls each of an impervious ceramic material joined one to the other around the periphery thereof to make a sealed enclosure;

sealed within the enclosure, a heating element constructed from a flexible. paper-like sheet of graphite or graphitic carbon;

lead means to supply electrical power to the heating element; and

port means by which purging inert gas can be supplied to, and withdrawn from, the sealed enclosure.

2. A heater unit as claimed in Claim 1 which is a tubular device.

3. A heater unit as claimed in Claim 2 which is a hollow tubular device in which material to be heated thereby can be passed along the middle of the tube.

4. A heater unit as claimed in any of the preceding Claims, wherein each wall is an elongate tube, one smaller in section than the other, the two being disposed one co-axially within the other to form an elongate annular enclosure within which the element is disposed.

5. A heater unit as claimed in Claim 4, wherein the tube ends are sealed, inner to outer, with end caps to provide the desired sealed enclosure.

6. A heater unit as claimed in Claim 5, wherein each end cap is sealed to the outer tubular wall, and there is provided cooling of the end cap adjacent the seal to prevent the seal overheating and being destroyed.

7. A heater unit as claimed in any of the preceding Claims, wherein the enclosure walls are made of re-crystallised alumina.

8. A heater unit as claimed in any of the preceding Claims, wherein the graphite-based heating element has thereover a thin coating or layer of a nitride (or other ceramic).

9. A heater unit as claimed in any of the preceding Claims, wherein the heating element for a tubular unit takes the form a helix, wound around the inner tube as a former, or of a plurality of linear bars, either running axially parallel with or circumferentially of the tube.

10. A heater unit as claimed in any of the preceding Claims, wherein the lead means provided to supply electrical power to the heating element are suitable electrical connections from the outside to the element within, the connections being of some suitable material that has a similar thermal coefficient of expansion.

11. A heater unit as claimed in any of the preceding Claims, wherein the port means by which the purging inert gas can be supplied to, and withdrawn from, the sealed enclosure, takes (for a tubular unit with end caps) the form of pipes sealed into and through the sealing end caps, and connectable to a source and a drain of the gas.

12. A heater unit as claimed in any of the preceding Claims and substantially as described hereinbefore.

13. A furnace whenever fitted with a heater unit as claimed in any of the preceding Claims.

Patents Act 1977
Ex: iner's report to the Comptroller under Section 17 (The Search Report)

Application number
9117221.3

Relevant Technical fields

(i) UK Cl (Edition K) H5H; HAA3, HAX2

(ii) Int CL (Edition 5) H05B 3/44, 3/62, 3/64

Search Examiner

R W BALDOCK

Databases (see over)

(i) UK Patent Office

(ii) ONLINE DATABASES WPI, CLAIMS

Date of Search

15 JULY 1992

Documents considered relevant following a search in respect of claims 1-14

Category (see over)	Identity of document and relevant passages	Relevant to claim(s)
X	WO 89/04108 (ELEC. DE FRANCE)	1,2,8,10, 11,14
X	US 5065131 (ELEC. DE FRANCE), English language equivalent of WO 89/04108, see especially colomn 1 lines 19-27, column 3 lines 37-58, column 5 lines 31-59	1,2,8,10, 14
X	US 4347431 (BELL TELEPHONE), See espcially column 3 lines 1-7, 28-31, column 6 lines 3-42	1-5,8-11, 14, 6
X	US 4135053 (ALCO STANDARD), See especially column 1 line 67 - column 2 line 23, column 2 line 67 - column 3 line 17	1,2,11,14 6
X	FR 2559886 (ELEC. DE FRANCE), See especially page 2 lines 15-19 and page 3 lines 16-19	1,2,7,11

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Category	Identity of document and relevant passages	Relevant to claim(s)

Categories of documents

X: Document indicating lack of novelty or of inventive step.

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A: Document indicating technological background and/or state of the art.

P: Document published on or after the declared priority date but before the filing date of the present application.

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